



Cobalt hexacyanoferrate electrodeposited on electrode with the assistance of laponite: The enhanced electrochemical sensing of captopril



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ABSTRACT

In this work, Co²⁺ ions were initially intercalated into the layers of laponite and then cobalt hexacyanoferrates (CoHCFe) were electrogenerated on the surface of laponite-Co²⁺ modified ITO via square wave voltammetry (SWV). At scan rate of 10 mV s⁻¹, the electrochemical response of the ITO/laponite/CoHCFe showed two pairs of well-defined redox peaks located about 0.44 and 0.672 V vs.SCE, attributed to the redox reaction of Co^{II}/Co^{III} and Fe^{II}/Fe^{III}, respectively. The modified electrode was highly electrocatalytic to the captopril oxidation. Moreover, the ITO/laponite/CoHCFe exhibited an enhanced analytical performance for amperometric detection of captopril with two enlarged linear ranges from 3.0 × 10⁻⁷ to 1.27 × 10⁻⁴ M and 1.63 × 10⁻⁴ to 3.76 × 10⁻⁴ M and a low detection limit (0.29 μM). The modified electrode was used for electrocatalytic determination of captopril in some real samples.

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1. Introduction

Captopril, with the chemical name of (1-[(2S)-3-mercapto-2-methyl propionyl]-L-proline), is the first orally active angiotensin converting enzyme inhibitor [1]. It is a well-known medicine applied to the treatment of hypertension and congestive heart failure [2]. During the metabolic pathway it is converted to a disulfide compound, which is eliminated together with unchanged captopril (40~60%) in urine. Determination of captopril has previously been reported by spectrophotometry [3], capillary electrophoresis [4], iodimetry [5], high performance liquid chromatography-mass spectrometry [6], high performance liquid chromatography [7], and FT-Raman spectroscopy [8]. These techniques possess some drawbacks, such as cost, sampling time, complexity, and interferences.

Reported literature has demonstrated that captopril could be electrochemically oxidized at the surface of different electrodes because of its thiol group [9,10]. There are many electrochemical methods, such as square wave voltammetry on static mercury drop electrode [11] and differential pulse voltammetry on modified carbon-paste electrode [12], have been used for its determination. In this work, electrocatalytic oxidation of captopril was developed.

It is an efficient method to expedite the electrochemical reaction of captopril.

Electrochemical detection provides a simple, low cost and highly sensitive approach to the analysis of a wide range of drugs [13,14]. However, this approach is sometimes restricted by limitations of selectivity due to the interference from the other redox active species which may undergo electrolysis at similar potentials to the target species in the solution. Thus, it is of great significance to develop a novel platform with high electroactivity and great selectivity.

Prussian blue (PB) and its analogues, have attracted growing interest on account of their interesting properties, for example electrochromicity [15], ion-exchange selectivity [16,17], capability of storing ionic charges [18,19] and ability to mediate electrochemical reactions [20,21]. Among the various PB analogues, cobalt hexacyanoferrate (CoHCFe) with an open and zeolite-like structure has better properties than other metal hexacyanoferrates because of its unique chemical and electrochemical properties. In CoHCFe, both cobalt and iron have two oxidation states and redox couples of (II) and (III), leading to a multitude of compound stoichiometries, redox states and unique electrocatalytic and electrochromic properties, reversible photoinduced magnetization and thermochromism behavior in a wide range of temperatures [22].

Herein, we try to construct a novel sensing platform based on small sized CoHCFe electrodeposited on electrode with the assistance of laponite. Laponite (hydrous sodium lithium

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magnesium silicate) is a form of layered aluminosilicates. The structure of the laponite is similar to the natural smectite clay hectorite which is an entirely synthetic crystalline layered silicate colloid with crystal structure [23]. Individual particles are disk-shaped crystals with 25 nm in diameter and 1 nm in thickness. Each sheet of octahedrally coordinated magnesium oxide is sandwiched between two layers of tetrahedrally coordinated silica in a single crystal. An isomorphous substitution of magnesium by lithium atoms gives rise to negative charge on its surface, which is counterbalanced by the positive charge of the sodium ions present in the interlayer. Sodium ions are leached which leads to a net negative charge on its surface in aqueous solution. Laponite can work as a cation exchanger. Thus, in this work, Co^{2+} ions were initially incorporated into the host laponite by intercalation method. Then, the CoHCF film was obtained by applying SWV to laponite- Co^{2+} -modified ITO electrode in a solution containing hexacyanoferrate (III). During this electrochemical process, the in situ growth of CoHCF crystallites is achieved. The as-synthesized CoHCF was nanosized, redox active and highly electrocatalytical to captopril.

2. Experimental Section

2.1. Materials

Laponite was obtained from Rockwood Specialities Inc. (Princeton, NJ). Cobalt chloride (CoCl_2), potassium hexacyanoferrate (III) ($\text{K}_3\text{Fe}(\text{CN})_6$) were purchased from local Sigma-Aldrich Chemical Co. (Shanghai, China). Captopril was purchased from local J&K Chemical Co. Captopril tablets (Beijing Jingfeng Pharmaceutical Co., Ltd., 25 mg captopril per tablet) were purchased from Jiuhetang Chinese Medicine drug store in Nanjing and the captopril solution was prepared as previous report [12]. The ITO plates (1.1 mm sildes with $6\text{--}8\ \Omega/\text{sq}$ surface resistivity, and $\geq 84\%$ transmittance) were purchased from Shenzhen Huananxiang Technology Co., Ltd. (Shenzhen, China). Ultrapure water obtained from a Millipore water purification system ($\geq 18\ \text{M}\Omega\ \text{cm}$, Milli-Q, Millipore) was used in all assays. All other reagents were of analytical grade and were used as received without further purification.

2.2. Apparatus

A CHI 660D electrochemical workstation (CHI Co., USA) was used for Square Wave Voltammetry (SWV), cyclic voltammograms (CVs) and chronoamperometry. All electrochemical studies were performed with a conventional three electrode system. Bare and modified ITO substrates with a size of $1 \times 1\ \text{cm}$ were used as working electrodes. A saturated calomel electrode (SCE) and a Pt wire electrode were used as reference and counter electrodes, respectively. The morphology of the modified electrode was investigated with a XL-30E scanning electron microscope (SEM) and the transmission electron microscopy (TEM) was performed on a Philips TECNAI-12 instrument. Attenuated total reflectance–Fourier transformation infrared (ATR-FTIR) spectra of samples were obtained with an IR-Prestige-21 FTIR spectrometer (Shimadzu Co., Japan). The X-ray photoelectron spectroscopy (XPS) experiments were carried out on K-Alpha (Thermo Fisher Scientific Co., USA). The Gaussian-Lorentzian distribution was used for fitting the spectra for each peak, in order to determine the binding energy of the core levels of the different elements.

2.3. Preparation of laponite- Co^{2+}

Co^{2+} was incorporated into the host laponite by intercalation method reported in the literature [24]: The laponite colloid

suspension ($3\ \text{mg mL}^{-1}$) was prepared by dispersing laponite in deionized water with stirring for 2 h in a water bath at $55\ ^\circ\text{C}$. After cooling to room temperature, 2 mL of 0.01 M cobalt chloride solution was quickly added to the 10 mL laponite colloid solution, under the condition of stirring. Then the mixture was stirred in a thermostated water bath at $25\ ^\circ\text{C}$ to be aged overnight and the final product was centrifuged and washed several times with deionized water and finally dried in air at room temperature.

2.4. Preparation of ITO/Laponite/CoHCFe

The obtained laponite- Co^{2+} nanoparticles were dispersed into deionized water with the concentration of $2\ \text{mg mL}^{-1}$. $50\ \mu\text{L}$ colloid solutions were spread on the surface of ITO electrode with a size of

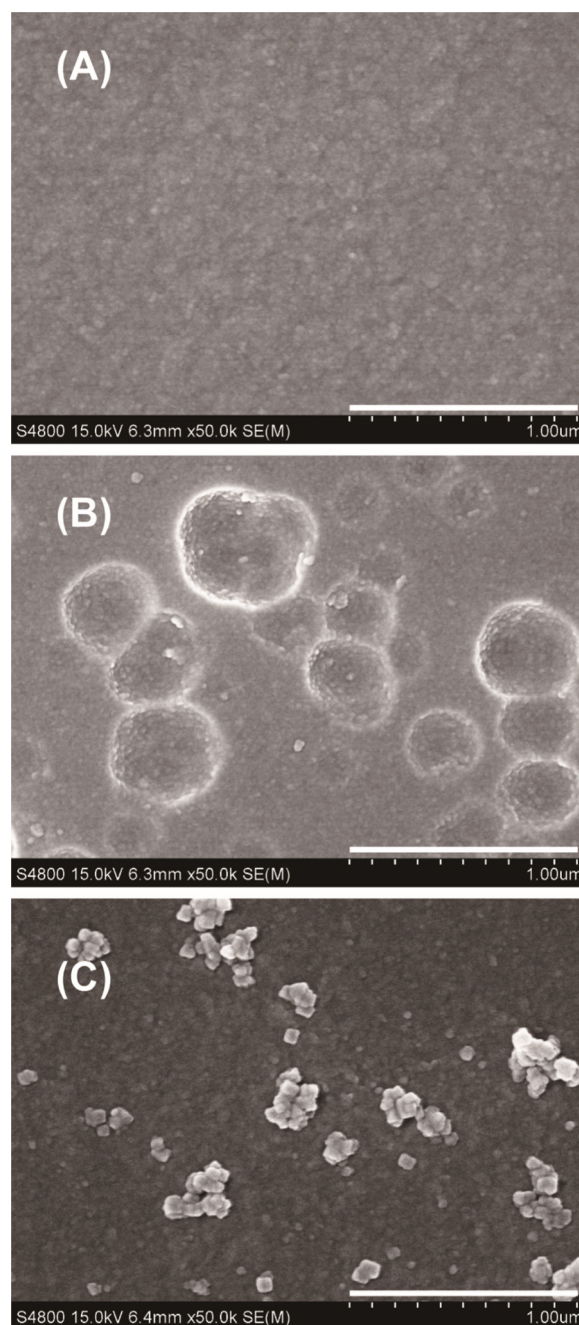


Fig. 1. SEM images of (A) ITO/laponite, (B) ITO/laponite- Co^{2+} and (C) ITO/laponite-CoHCFe.

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